

Method of Test for
**PERCENT LEAD (Pb) IN PAINT CHIPS VIA INDUCED COUPLED
PLASMA (ICP) SPECTROSCOPY**
DOTD Designation: TR 529-10

I. Scope

This method describes the determination of percent lead in paint chips collected from various structures.

II. Apparatus

- A. **Analytical balance** – 120g capacity with accuracy to $\pm 0.0001\text{g}$
- B. **Evaporating dish** – 100mL
- C. **Volumetric flask** – 100mL
- D. **Muffle furnace** – capable of maintaining a temperature of $410 \pm 10^\circ\text{C}$
- E. **Induced Coupled Plasma Spectrometer (ICP)**
- F. **Separation funnel** – with #40 filter paper
- G. **Hot Plate** – capable of maintaining a temperature of $100 \pm 5^\circ\text{C}$
- H. **Beaker** - 250 ml
- I. **Dessicator**
- J. **Weighing paper**
- K. **Tweezers**
- L. **Deionized Water**
- M. **Paint Chip Worksheet**, Figure 1

III. Reagents

- A. **Nitric acid solution** – Dissolve concentrated nitric acid into deionized water in a ratio of 1:3 respective to nitric acid and water.
- B. A solution of lead with a concentration accurate to 10 ppm to create calibration curve.

IV. Health Precautions

Proper equipment and precautions are to be used whenever toxic samples are used. **It is fair to assume if paint samples are being tested for lead, the samples should be handled as though they contain lead.** Use appropriate safety equipment such as safety glasses, gloves, and dust masks designed to filter lead particulates. Wash hands frequently. Standard sample handling protocol should be adhered to, both prior to and after testing. Spent samples should be handled as though they contain lead and properly stored until final disposition.

V. Sample

The sample shall be obtained from paint chips on structural steel. It should be a representative sample of the primer, intermediate, and topcoat of the paint system collected at a minimum of 3 different areas of the structure. Take care to ensure that the sample consists of only the paint system, not the steel substrate when scraping. A minimum of 5 grams is needed.

VI. Procedure

- A. Tare the weighing paper. Use tweezers to place 0.2 g to 0.3 g of paint chips on the weighing paper. Weigh to the nearest 0.001 g and record weight on the worksheet.

- B. Place weighed test specimen on evaporating dish.
- C. Place evaporating dish containing test specimen in a preheated muffle furnace set at $410 \pm 10^\circ\text{C}$.
- D. Allow sample to remain at 410°C for 2 hours.
- E. Turn off muffle furnace.
- F. Let muffle furnace cool for 1 hour.
- G. Remove evaporating dish containing sample and place it in a desiccator for 30 minutes.
- H. Using the 250ml beaker, dissolve the sample in approximately 40 ml of a 1:3 nitric acid/deionized water solution on a preheated hot plate set at medium temperature.
- I. Allow sample to digest for at least 30 minutes on the hot plate.
- J. Filter acid solution into 100 ml volumetric flask.
- K. Dilute with deionized water until the meniscus reaches the 100 ml allowing the bottom of the meniscus to mark on the volumetric flask.
- L. Repeat steps A thru K two more times for a total of 3 test specimens.
- M. Perform analysis on calibrated (ICP) against lead standard and record the concentration of lead in parts per million (ppm) on the worksheet.

VII. Calculations

$$\% \text{Lead} = \frac{A}{W \times D} \times 100$$

where,

A = lead concentration in (ppm)
obtained from the ICP

W = weight of the sample in grams

D = dilution factor of 10000 (ppm/g)

100 = constant (for percentage)

example:

$$A = 9.6 \text{ ppm}$$

$$W = 0.3000 \text{ g}$$

$$D = 10000 \text{ ppm/g}$$

$$\% \text{Lead} = \frac{9.6}{0.3000 \times 10000} \times 100$$

$$= 0.0032 \times 100$$

$$= 0.32 \%$$

VIII. Report

The data shall be reported to the nearest hundredth percent lead on the paint chip worksheet, Figure 1.

IX. Normal Test Reporting Time

The normal amount of time to report on lead content analysis is approximately 2 days.

X. Additional Information

The equation can be derived from the following information:

$$\% \text{ lead} = \frac{A \times \left(\frac{\text{mg/L}}{\text{ppm}} \right) \times \frac{\text{g}}{10^3 \text{ mg}}}{\left(\frac{\text{Sample (g)}}{100 \text{ mL}} \right) \times \left(\frac{10^3 \text{ mL}}{1 \text{ L}} \right)} \times 100\%$$

This equation can be simplified to state:

$$\% \text{ lead} = \frac{A \times \left(\frac{\text{g/L}}{10^3 \text{ ppm}} \right)}{\left(\frac{\text{Sample (g)} \times 10^3}{100 \text{ L}} \right)} \times 100\%$$

$$\% \text{ lead} = \frac{A}{\text{Sample} \times 10000 \text{ ppm/g}} \times 100\%$$

where A is parts per million (ppm) of Lead determined by the Induced Coupled Plasma Spectrometer (ICP).

Louisiana Department of Transportation and Development Materials & Testing Section		8/97
PAINT CHIPS (TR 529)		
REMARKS 2 <div style="border-bottom: 1px solid black; height: 20px; width: 100%;"></div> <div style="border-bottom: 1px solid black; height: 20px; width: 100%;"></div>	<div style="text-align: center;">TEST RESULTS (Max. of 15 Characters)</div> <div style="border-bottom: 1px solid black; height: 20px; width: 100%;"></div> <div style="text-align: center;">P/F</div>	
TYPE	<div style="border-bottom: 1px solid black; height: 20px; width: 100%;"></div> <div style="border-bottom: 1px solid black; height: 20px; width: 100%;"></div>	
LEAD % (A x DF) / (SW x 10000)	<div style="border-bottom: 1px solid black; height: 20px; width: 100%;"></div> <div style="border-bottom: 1px solid black; height: 20px; width: 100%;"></div>	
Parts Per Million of Lead (A) Dilution Factor (DF) Sample Weight, g (SW)	<div style="border-bottom: 1px solid black; height: 20px; width: 100%;"></div> <div style="border-bottom: 1px solid black; height: 20px; width: 100%;"></div> <div style="border-bottom: 1px solid black; height: 20px; width: 100%;"></div>	
Tested By: <u>TAT</u> Date: <u>1/6/10</u> Checked By: _____ Date: _____		
APPROVED By: <u>RC</u> Date: <u>1/6/10</u>		
09/ <div style="border-bottom: 1px solid black; height: 20px; width: 100%;"></div> /620/ <div style="border-bottom: 1px solid black; height: 20px; width: 100%;"></div> / N		

Figure 1
Paint Chip Worksheet